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13. ABSTRACT (Maximum 200 words) In this program we developed an important class of multifunctional and self assembling materials based on the use of triblock molecular structures that had both luminescent chromophores and dipole moments. The objective was to integrate self assembly, encoded in the triblock structure, luminescent properties, and the properties characteristic of materials that have macroscopically polar structure. Our research over the past few years demonstrated self assembly in these materials with the formation of layered nanostructures measuring a few nanometers, and also photoluminescent behavior in the solid state of these materials. We also found these materials form thin films on substrates such as glass that exhibit spontaneous second harmonic generation when probed with infrared laser beams. An important milestone in the program was the discovery of piezoelectricity in the self assembling materials containing triblock molecules composed of oligostyrene, oligoisoprene, and biphenyl ester-phenylene vinylene blocks. The experiments were performed in a Nomarski interferometer, and a piezoelectric coefficient of 0.028 pm/volt was observed in thin films that were never exposed to an electric field. We concluded from this observation that the observed signal is the result of spontaneous polar organization by self assembly. The multifunctional materials were also found to have the unusual property of having almost identical absorption spectra in both dilute solution as well as the solid state with only a relatively small blue shift of 9 nm. A narrow red shifted emission was observed also which is not common in similar materials without self assembling properties. We believe this behavior is connected to the nanostructured nature of these materials. Finally, the program started an investigation of materials that could self assemble in aqueous media and were therefore designed to have amphiphilic nature.					
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1. List of Manuscripts:

- G. N. Tew, M. U. Pralle, and S. I. Stupp: Supramolecular Materials with Electro- Active Chemical Functions, *Angewandte Chemie*, 112, 3, 527 (2000).
- M. U. Pralle, K. Urayama, G. N. Tew, D. Neher, G. Wegner, and S. I. Stupp: Piezoelectricity in Polar Supramolecular Materials, *Angewandte Chemie*, 112, 8, 1546 (2000).
- S. I. Stupp, L. Li, M. U. Pralle, M. Sayar, G. N. Tew, and E. R. Zubarev: Self-Assembly of Organic Nano-Objects into Functional Materials, *MRS Bulletin*, 25, 4, 42 (2000).
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- G.N. Tew, L. Li, and S. I. Stupp, "Polar and Luminescent Supramolecular Films, *J. Am. Chem. Soc.* 120, 5601 (1998)
- G.N. Tew, and S. I. Stupp, "Multifunctional Supramolecular Materials", *ACS Symposium Series: Functional Polymers*, A. O. Patil, D. N. Schulz, and B. Novak, eds., 218 (1998).
- S. I. Stupp, M. Keser, G. N. Tew, "Metastability in Polymer Phases and Phase Transitions", *Mat. Res. Soc. Symp. Proc.*, Fall Meeting, Boston (1997).
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2. Scientific personnel

Gregory N. Tew

Martin U. Pralle

Leming Li

3. Report of inventions

None

4. Scientific progress and accomplishments

We have observed piezoelectricity in a self assembling supramolecular material. The polar self assembly of nanostructures results in a layered solid that is capable of electromechanical transduction.

5. Technology transfer

None

Multifunctional Materials Based on Self Assembly of Molecular Nanostructures

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Submitted to

Douglas J. Kiserow

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by

Samuel I. Stupp

Department of Materials Science and Engineering
Department of Chemistry
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Northwestern University

April 2001

Description of the research problem

One of our objectives was to develop supramolecular materials by self assembly that are multifunctional combining for example mechanical, adhesive, photonic, electronic, and sensing functions. We were able to establish some of the rules that can lead to the rational design of new molecular precursors to create functional supramolecular materials from triblock rodcoil molecules. The rod segments in these triblock molecules are chemically identical and are thermodynamically driven to aggregate into nanocrystals because of the frustration of three dimensional crystallization by the chemically diverse oligostyrene-co-oligoisoprene blocks. The free energy balance between crystallization of rod segments and steric forces in coil segments affecting entropy is believed to be responsible for the formation of nanostructures. In our laboratory, we have discovered that these molecules create novel self assembling materials with unexpected and interesting properties.

The specific focus of this research was the synthesis of electronically and photonic active supramolecular nanostructures. These materials are of interest for electroluminescent or color switchable coatings, third order photonics, piezoelectric transducers, and other field responsive properties that could be achieved by self assembly. Toward the end of the program we turned our attention to a series of novel amphiphilic molecules in which hydrophobic segments are based on branched phenylene vinylene, and the hydrophilic segments contain amino acid and/or sugar residues. Eventually, structures of this type which should self assemble in aqueous media may be useful in detection of biological organisms electronically.

Summary of the most important results

Synthesis of Multifunctional Self Assembling Molecules

We completed the synthesis of two novel rodcoil molecules containing phenylene vinylene segments **4** and **5** (figure 1), and also worked on structures **1-3** in figure 1. The triblock rodcoil molecules were characterized by methods such as $^1\text{H-NMR}$, GPC, TEM, SAXS, and polarized optical microscopy. SAXS experiments revealed self organization of **1** into films containing layers 6.9 nanometers (nm) thick. Variable temperature SAXS indicates these layers thicken with increasing temperature. Several curves are shown in Figure 2, demonstrating the layer spacing grows as the temperature is increased. At 100°C , the layer spacing has reached its maximum of 7.7 nm and further increases in temperature do not result in an increase in layer spacing. Interestingly, when the temperature is increased to 125°C , a new peak at a d-spacing of 4.5 nm is observed. The top trace in Figure 2 at 200°C demonstrates the thermal stability of order in this material. We assigned the new peak to the ordering of supramolecular units which have dimensions of 4 to 5 nm in cross section and their organization into larger domains at elevated temperatures.

TEM and electron diffraction of thin films of **1** revealed nano-sized domains containing strongly scattering materials (see figure 3 inset) and indicates the crystalline nature of the observed nano-sized aggregates. The areas which appeared as a result of phase and diffraction contrast are believed to contain aggregates of the rod-like segments. The rod segments are the only blocks in the chemical structure of **1** which could crystallize, and lighter regions of the micrograph which transmit electrons must contain amorphous material. The average diameter of the aggregates is roughly 2nm

and the micrograph indicates some variation in the size of aggregates. Interestingly, the diffraction pattern of **1** consists of small arcs indicating a preferred crystalline orientation among the supramolecular units observed. Local orientation among individual nanostructures is remarkable and suggests some cooperative packing interactions among them. In addition, the diffraction patterns characterize the crystal structure of the rod domains within the supramolecular units as an orthorhombic unit cell with *a* and *b* lattice parameters equal to 18.72Å and 11.10Å, respectively.

Interestingly, when films of **1** are annealed near 125°C for several hours, TEM micrographs (figure 3) indicate the average size of the supramolecular units increases to roughly 5 nm and strip-like domains disappear. In addition, the electron diffraction pattern (inset figure 3) confirms the crystal structure, and orientation of rod domains of supramolecular units is preserved during annealing. Furthermore, the increase in aggregate size and observation of aggregates with 4 to 5 nm cross section is consistent with the observations in SAXS studies. Larger aggregates could be expected to increase the *d*-spacing of the layers, while objects 4 to 5 nm in cross section if ordered, may give rise to the observed X-ray maximum at 4.5 nm. We were able to ultramicrotome films of **1** (see figure 4) and discovered large layered domains with common orientation and a period of 7.5 nm in agreement with SAXS data. This observation suggests that bulk polar order emerges by self organization in the materials, as found for films containing chemically different molecules. This observation suggests the very important fact that bulk polar order could be observed in systems that vary chemically and could therefore integrate properties related to their local chemistry.

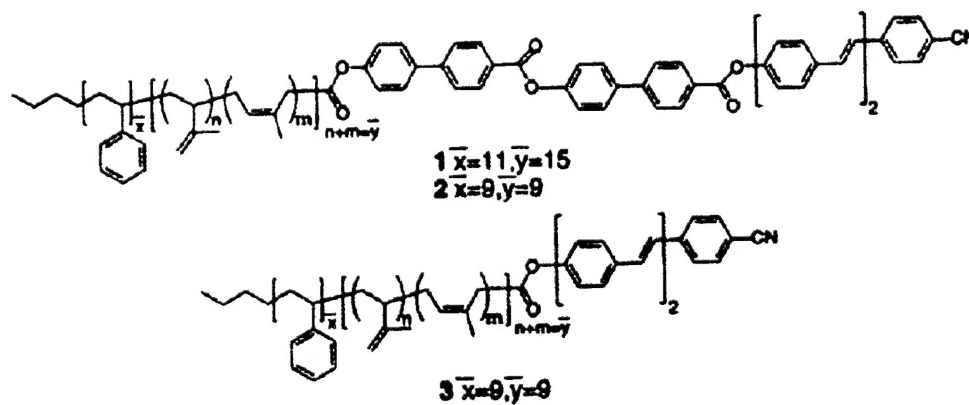
The ability to form a superlattice of supramolecular objects is expected to increase as the size and shape of the objects becomes more uniform. We inferred that a longer diblock coil would lead to a novel uniform population of molecules and possibly more regular nanostructures. Figure 5 shows two TEM micrographs which compare the unannealed supramolecular films of **1** and **2**. The TEM micrographs show that indeed the longer coil results in more uniform nanostructures and better orientation among nanostructures. However, we are still not certain of the reasons for the more uniformly sized nanostructures of **2**.

We completed the synthesis of electronically and photonicly active triblock structure **4** and **5**. Applying the rules we learned in previous work, **5** was designed to form mushroom shaped nanostructures and also deliver increased functionality. Preliminary data on films of **5** indicated the formation of polar monolayers and nanostructures formation. These molecules lead us to explore sensory properties in these self assembling materials. The structure of **5** contained triphenylamine blocks which oxidized easily under the influence of a small bias. The middle block could potentially form ionically conducting regions, and finally the rod segments exhibit photoluminescent behavior. Figures 6 and 7 show the final pathway we actually used to synthesize triblock structures **5**.

Luminescent Properties

As cast from solution some triblock rodcoil molecules self organize into polar macroscopic films. Such films should give rise to nonlinear optical properties and these have been investigated in the past resulting in the observation of strong second harmonic generation which scales with film thickness. Piezoelectric and electrostriction

experiments were carried out on material 1 (see structure below) to further probe the extent of polar order in these films. These experiments were carried out on a modified Nomarski interferometer with lock-in detection allowing for the independent measurement of the piezoelectric and electrostrictive coefficients.¹ Figure 8 is a plot of the piezoelectric response as a function of voltage and is an average of multiple scans to ensure reproducibility. The resultant piezoelectric coefficient (the slope of the linear fit) is 0.028 pm/V. This measurement was taken from a sample that was never exposed to an external electric field and therefore the signal can only arise from spontaneous polar organization by self assembly. The piezoelectric coefficient increased when the film was poled above the softening temperature.



In order to further investigate the self organizing properties of the film an experiment was carried out to monitor the net polarization as the sample is annealed at various temperatures. A plot of piezoelectric coefficient as a function of annealing temperature (figure 9) shows a change in piezoelectric response starting around 80°C that correlates with a molecular relaxation observed through dielectric spectroscopy. The observed increase in the piezoelectric response therefore suggests that about the relaxation temperature the system self organizes into a polar state. It is important to

note that upon cooling to room temperature the net polarized state remains, suggesting that the polar packing of nanostructures is thermodynamically favored.

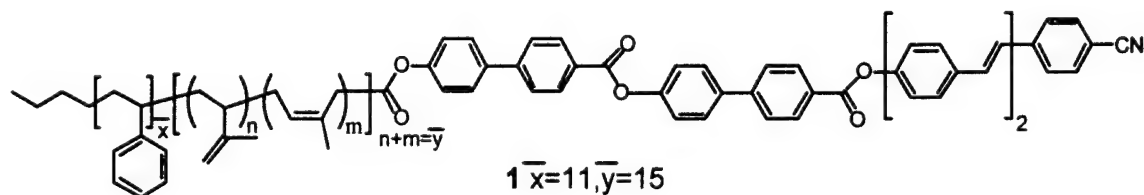
The florescent properties of these materials were also characterized and interesting observations were made. Figure 10 shows both the adsorption and emission spectra for **2** as a dilute solution or solid film. Interestingly, the absorption spectra corresponding to the dilutes solution and solid film overlap almost exactly with only a relatively small blue shift of 9 nm observed for the maxima. A similar small shift is observed in the emission spectra, the emission profile of solid **2** is narrower and red shifted by only 10 nm relative to the dilute solution profile. Furthermore, the red shift in solid state emission is smaller relative to other similar compounds reported in the literature ², while the decrease in width of the emission in the solid state is typically not observed. Yet, when the dilute solution and amorphous solid films of **3** were studied, the adsorption and emission spectra revealed shifts observed commonly in other PV-based materials. These spectra are shown in Figure 11. Comparing peak maxima, the solid films show substantial blue shifted absorption (31 nm) and red shifted emission (26 nm). As mentioned before, films of **3** were found to be amorphous by ED, and contain no detectable hierarchical ordering as determined by SAXS and optical microscopy.

There is a clear difference in the absorption and emission profiles for the supramolecular film of **2** and the unordered film of **3**. In contrast to the amorphous material, the absorption and emission of supramolecular films are very similar to those obtained from dilute organic solutions of the same material. The film in which self assembly is not observed exhibits more classical differences between the dilute solution

and the solid state. Both theoretical³ and experimental⁴ reports in the literature suggest small aggregates of two to six stilbene molecules can alter the absorption and fluorescence spectra of the system. In addition, Brédas and co-workers have shown theoretically that the angle between the long molecular axes of neighboring chromophores can have a dramatic effect on the absorption and emission profiles as well as the luminescence efficiency. It is likely that the observed spectra for the supramolecular film is a result of the specific packing interactions among chromophores within the mushroom-shaped aggregates. It is possible that within the nanostructures the chromophores are ordered into sub-domains that can not be observed by TEM or ED. This additional ordering within the nanostructures could produce smaller aggregates on the order to two to six molecules thus resulting in the observed spectra. It is our belief that the solid state spectra result from a combination of smaller aggregates within the mushroom shaped structure and a non-zero angle between the molecular axes of chromophores.

Polarization and Piezoelectricity by self assembly

Polarization experiments confirmed the presence of bulk polar order within our supramolecular materials. Figure 12 is a plot of the polarization response vs. temperature for a film composed of material **1** (see structure below).



Import

antly, this film was not annealed prior to the measurements such that changes in the film could be observed during the annealing process. A measurable polarization was

not detected in as cast samples, but above the relaxation temperature of 80 °C a positive polarization was observed. Further increases in temperature result in higher polarization and most importantly the net polarization is retained when the samples are cooled to room temperature. This thermally activated re-organization is clear evidence that a polar structure is indeed stable in this material. A sample poled in an electric field of 45 MV/m was annealed and the piezoelectric response in the absence of the poling field was monitored with increasing temperatures. Plotting piezoelectric coefficient and plate compressibility vs. temperature, one can observe the decay of piezoelectric response down to the nominal value for the unpoled sample. At the same time the softening of the material is observed as the temperature is increased. The fact that the piezoelectric signal returns to a similar value to that measured in a film annealed but never exposed to a poling field offers support for the possibility that net polarization in these systems is thermodynamically stable.

The dielectric spectroscopy data from a film of this same material is shown in Figure 13 and clearly shows a relaxation near 80 °C. Figure 14 is a plot of polarization vs. electric field for both positive and negative bias. The saturation values of polarization lie in the range expected for complete alignment of dipolar species. This observation is strong evidence for the polar nature of domains in this supramolecular material. The data shows a slight hysteresis with a remnant polarization similar to that observed just after annealing the film without an applied field. This suggests that the polar order is stabilized in the self assembled state, similar to what is observed in ferroelectric materials.

Synthesis of Branched Structures for Self Assembly in Water

The synthesis of phenylene vinylene dendrimers has been reported recently in the literature by two different laboratories, Yu and Meier. Our synthetic scheme employs a modified route similar to that of Yu's laboratory which does not require the presence of protecting group chemistry. Synthesis of generation 1 uses a Horner-Emmons procedure that we have taken advantage of in our laboratory over the last several years to form new *trans* vinyl bonds. This reaction leads almost exclusively to *trans* products (>95%). For the synthesis of generation 2 dendrons, the heck reaction is employed to form the necessary vinyl bond. This is a synthetic strategy that does not require protecting groups given the compatibility of chemical functions. For example, in this case the bromide function does not react during the first synthetic step (Horner-Emmons) but is reacted in the second step (Heck). Likewise, the aldehyde group is non-reactive during the Heck reaction. Scheme 1 outlines the synthesis of PV dendrons that are to be employed as the hydrophobic portion of the amphiphiles. The hydrophilic segments are synthesized using standard peptide chemistry to assemble generation 1 or 2 lysine dendrons (Scheme 2). Scheme 3 outlines the reactions necessary to connect the two halves of the amphiphile. An esterification reaction followed by deprotection of the amine groups results in formation of the amphiphiles composed of PV and lysine.

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4. M. W. Wagaman, R. H. Grubbs, *Macromolecules*, 30, 2978-3985 (1997)

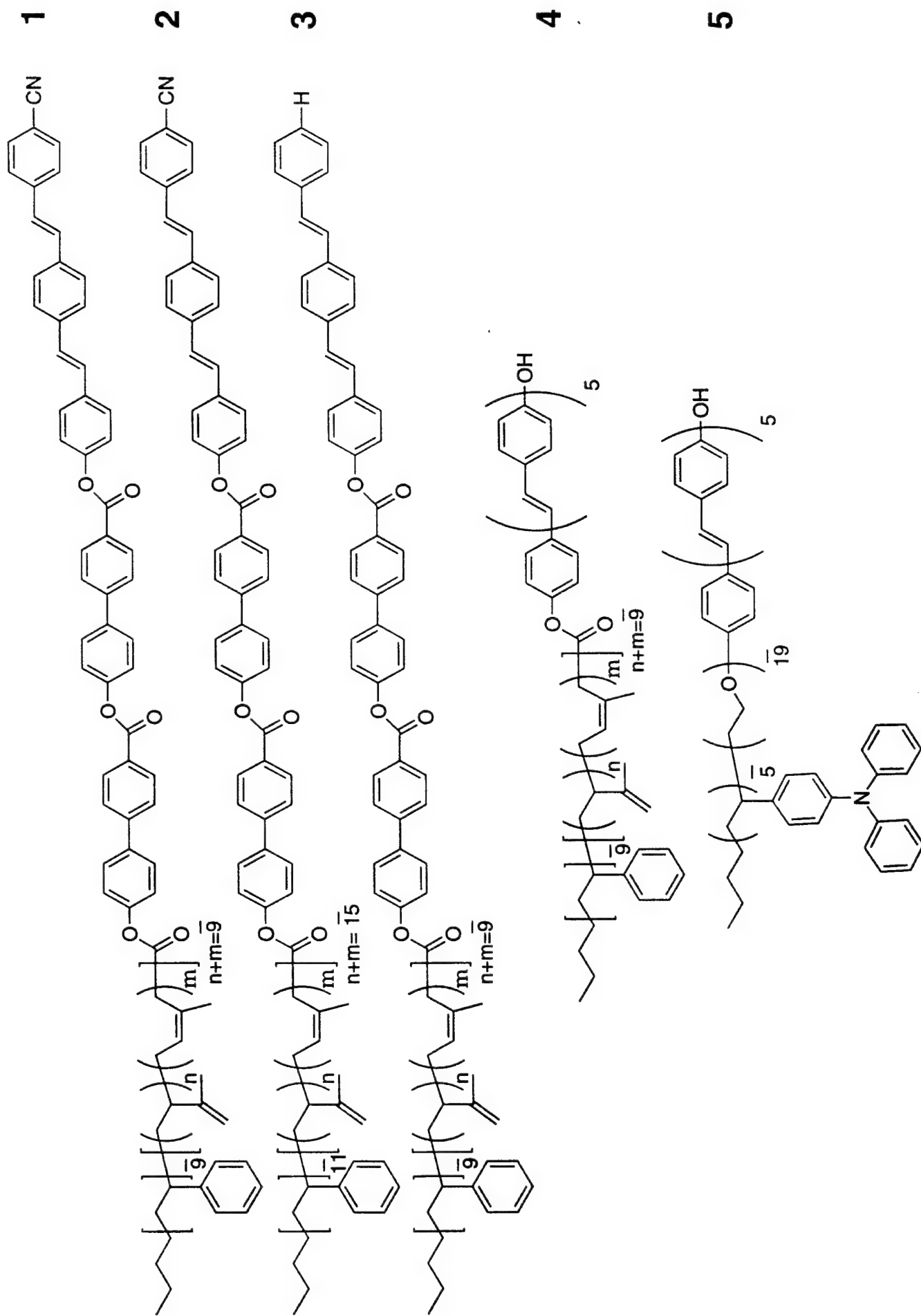


Figure 1. Electronically and photonically active self organizing triblock molecules

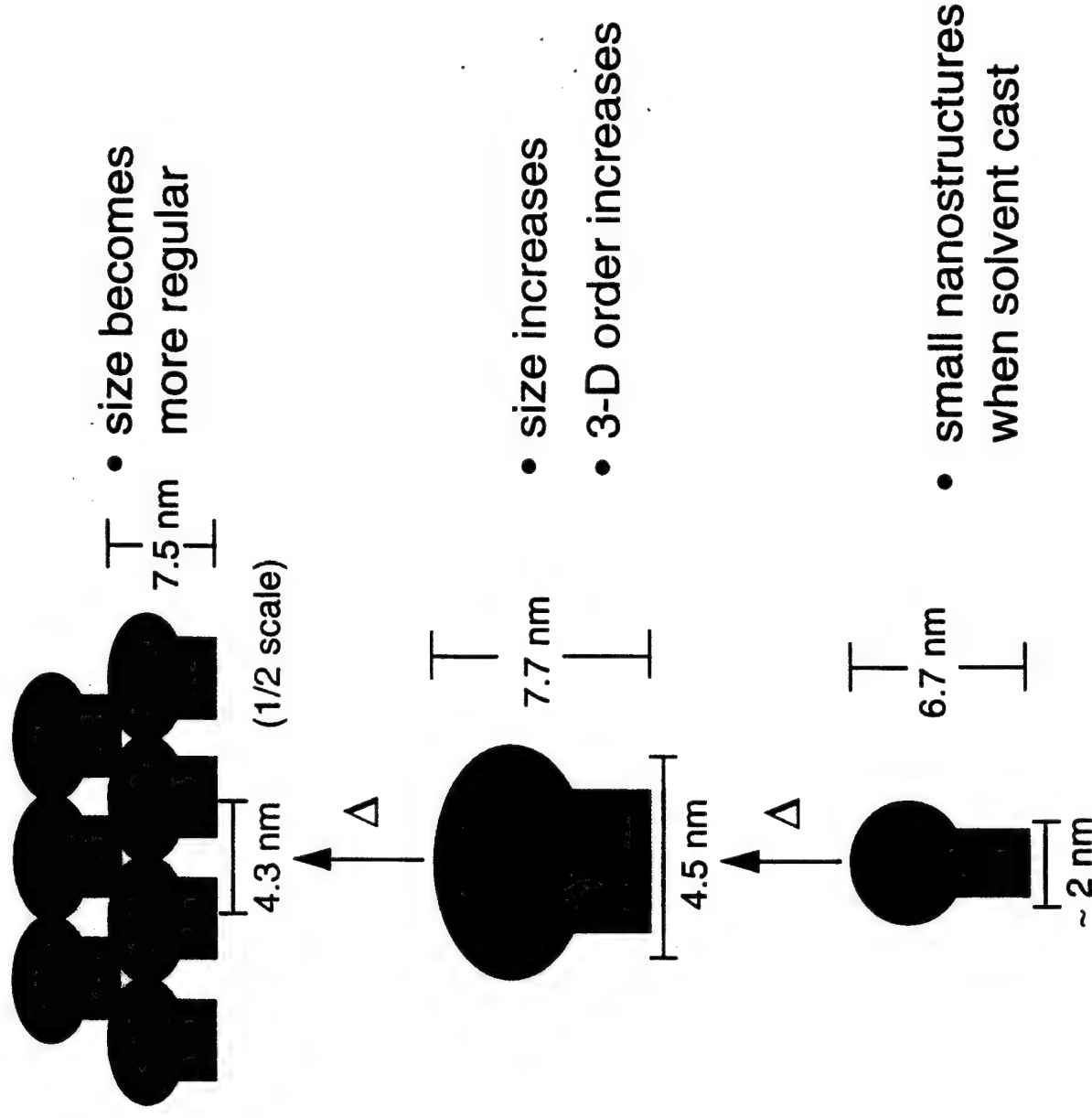
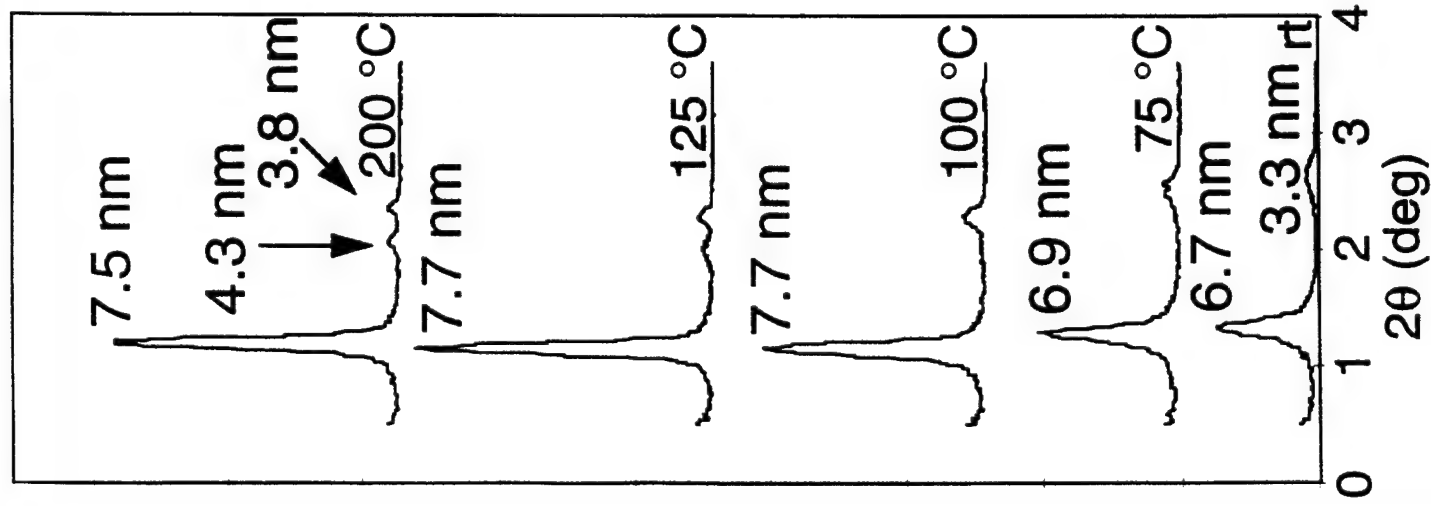
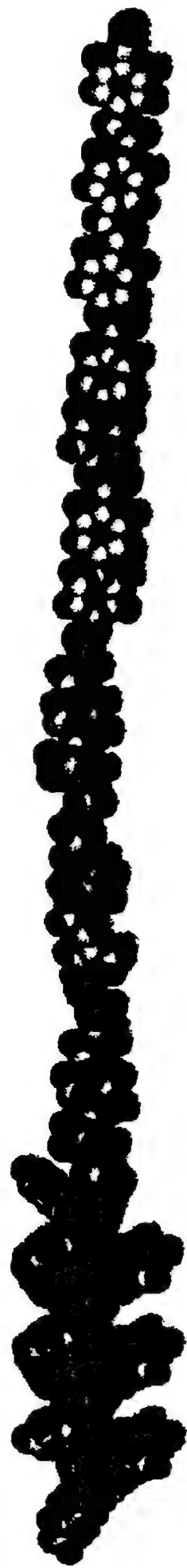
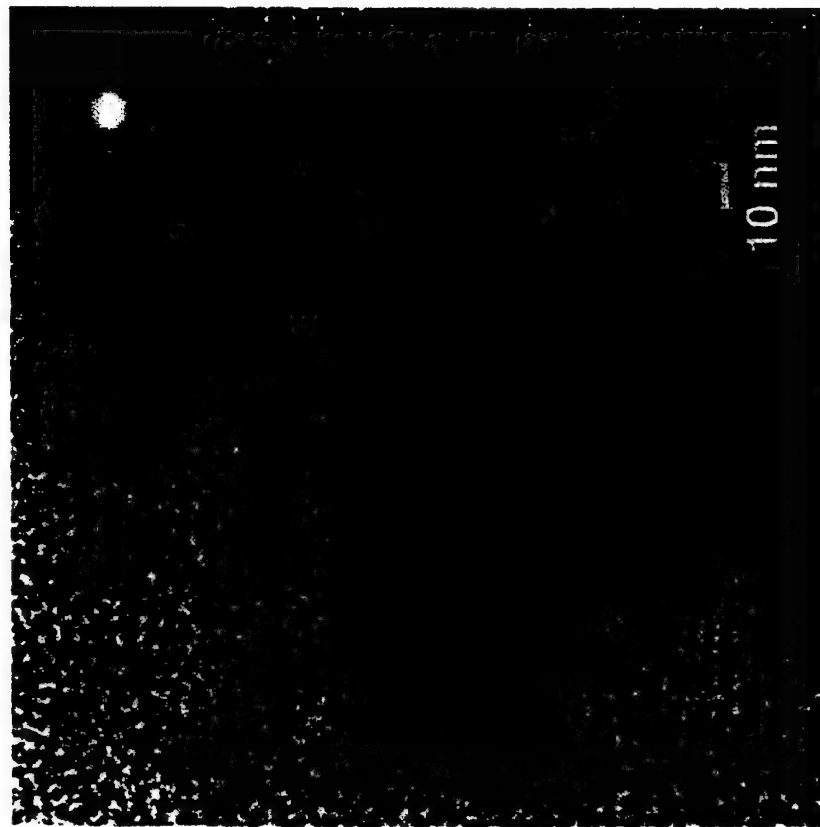


Figure 2. The molecular spacing and order within the material increases with increasing temperature



solvent cast

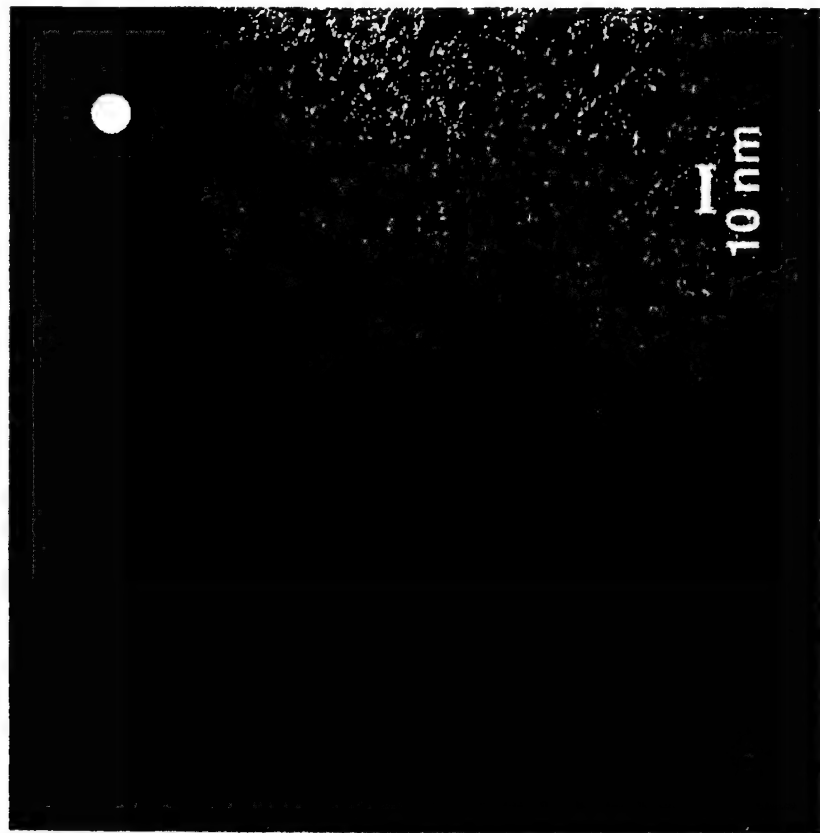


~ 2 nm aggregates

125 °C

2 h

annealed

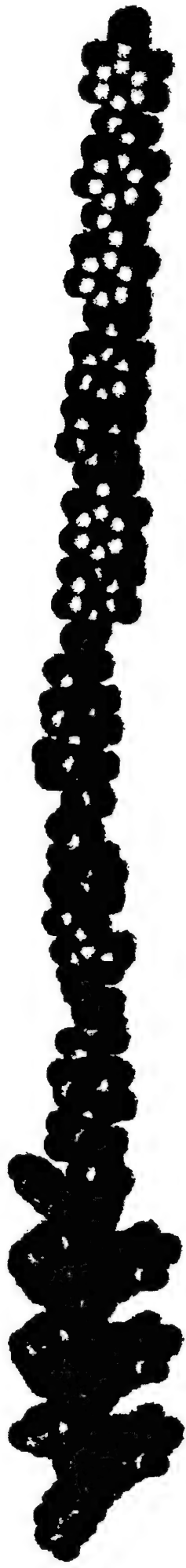


~ 5 nm aggregates

Figure 3. Diameter of supramolecular objects increase after annealing.



Figure 4. Ultramicrotomed sample of **1** indicates long range orientation within the supramolecular film.



9 styrene, 9 isoprene units



11 styrene, 15 isoprene units



Figure 5. Increasing the length of the coil leads to more uniform size and better orientation among nanostructures.

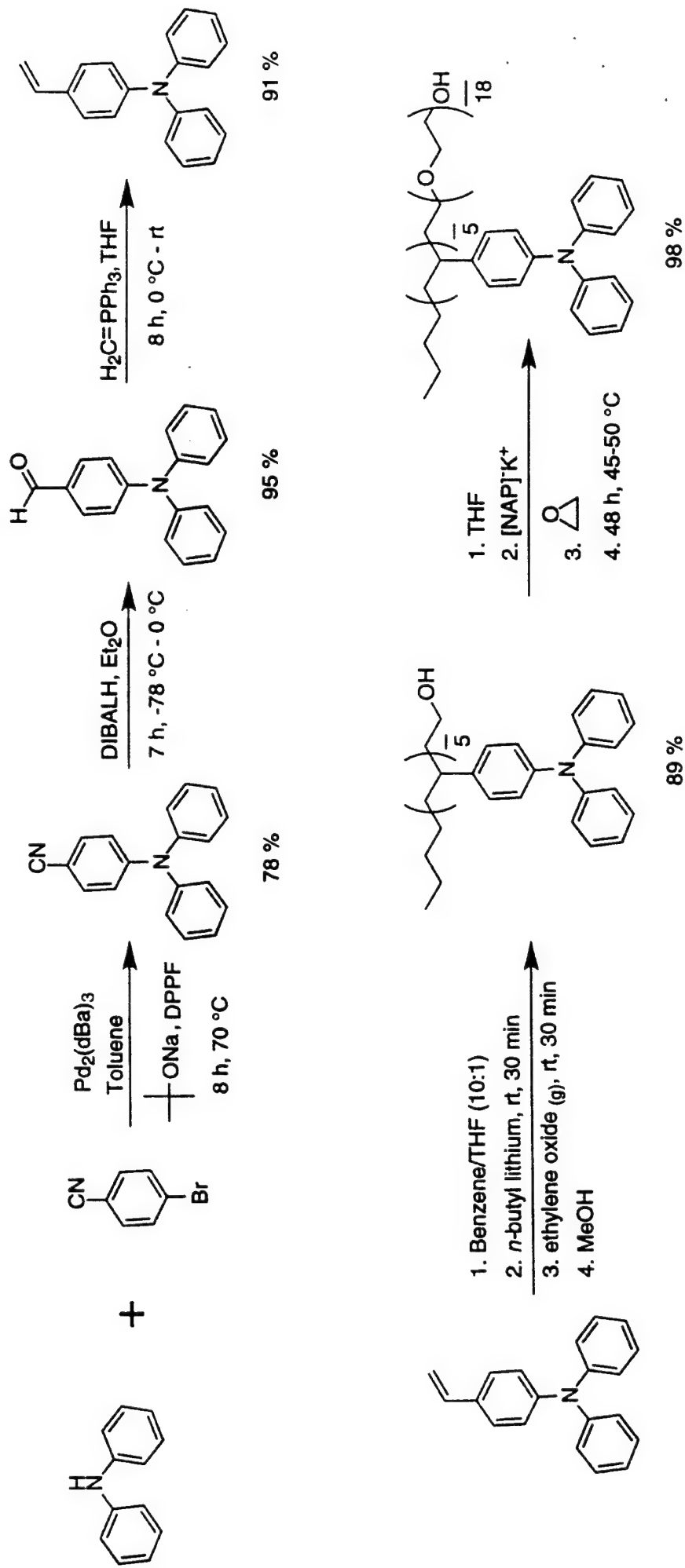


Figure 6. Synthetic route to an electronically and photoinitiationally active monomer and coil.

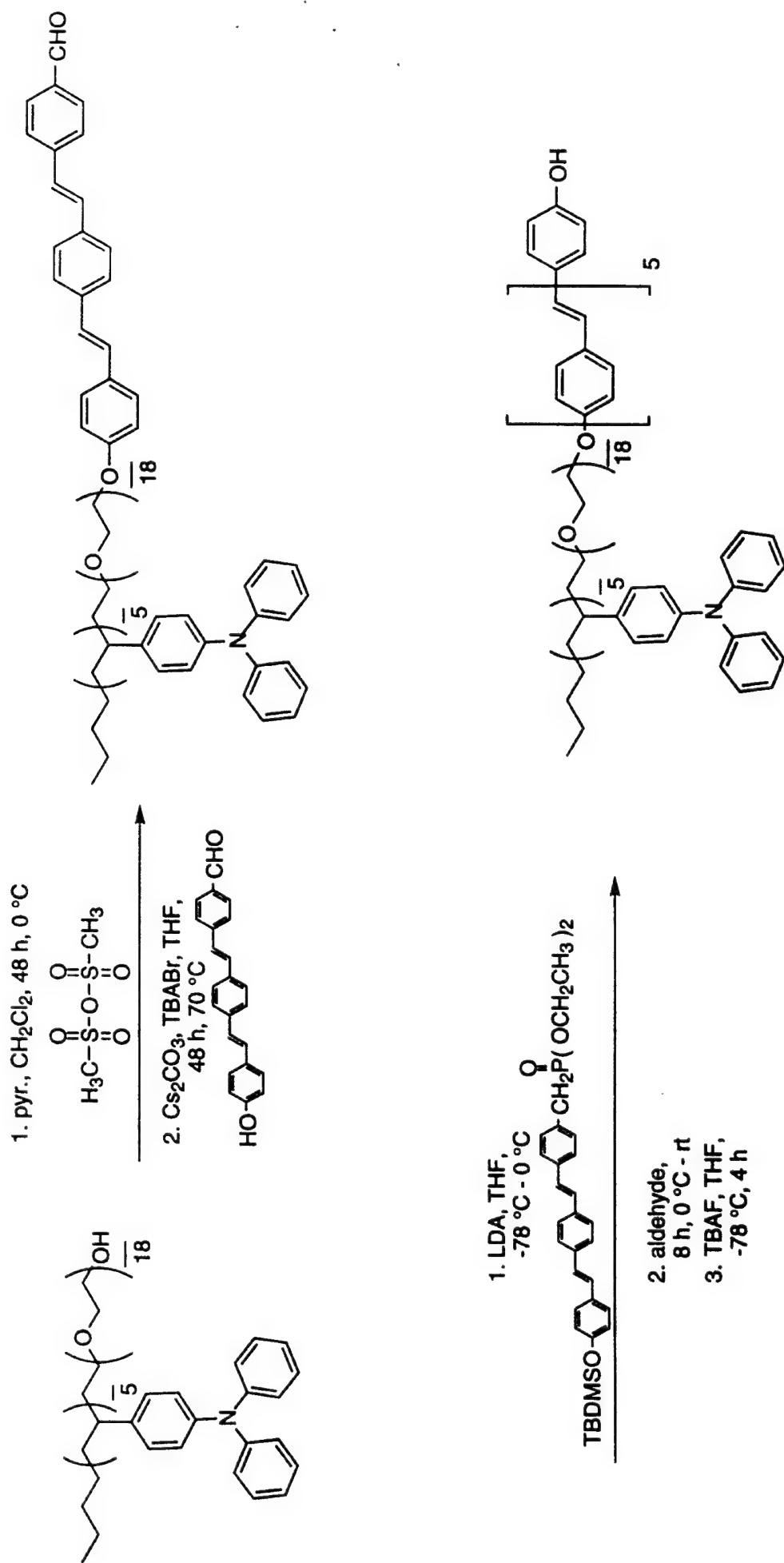


Figure 7. Synthetic pathway for electro-active triblock structure.

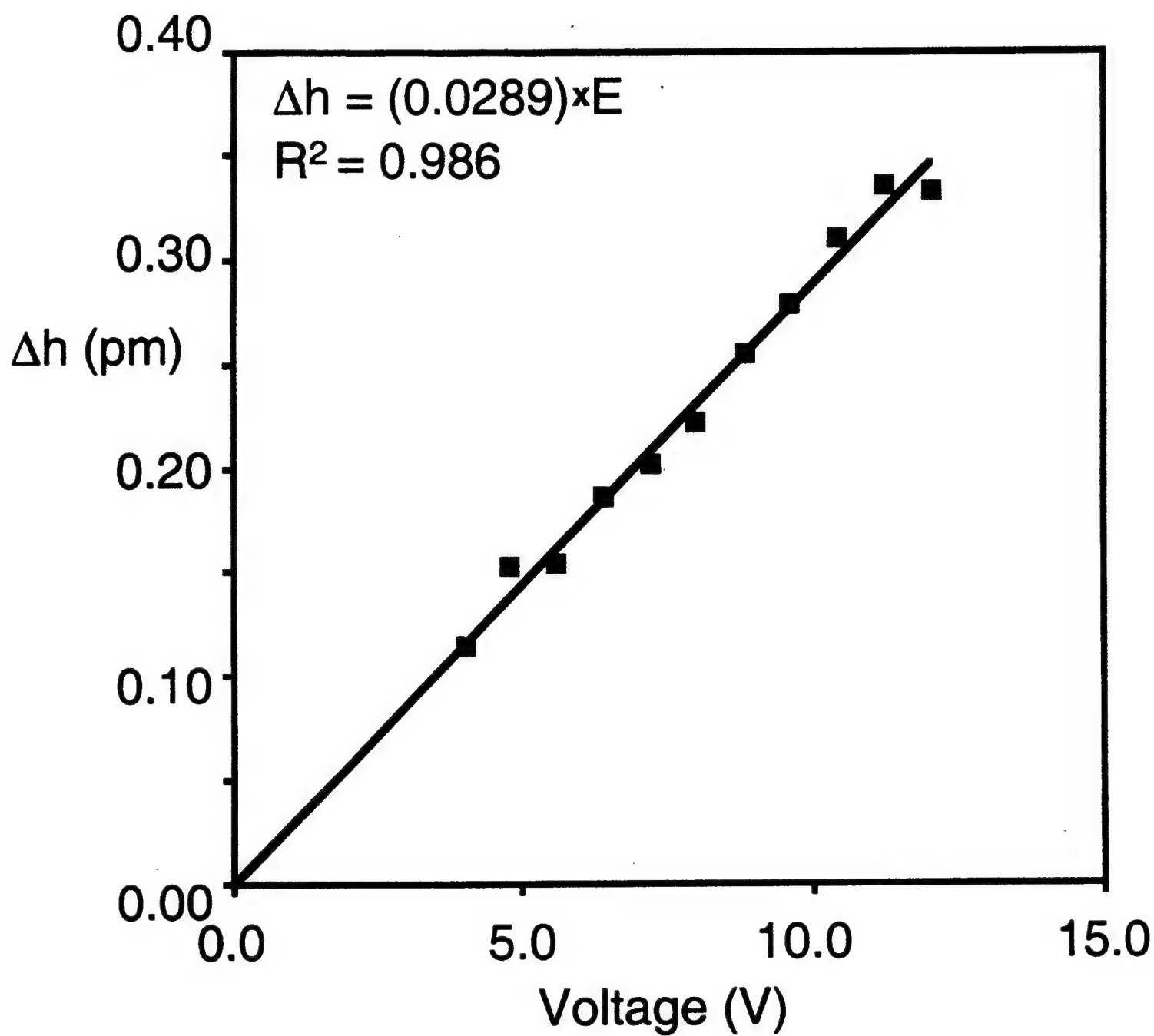


Figure 8. Dimensional changes as a function of applied voltage.

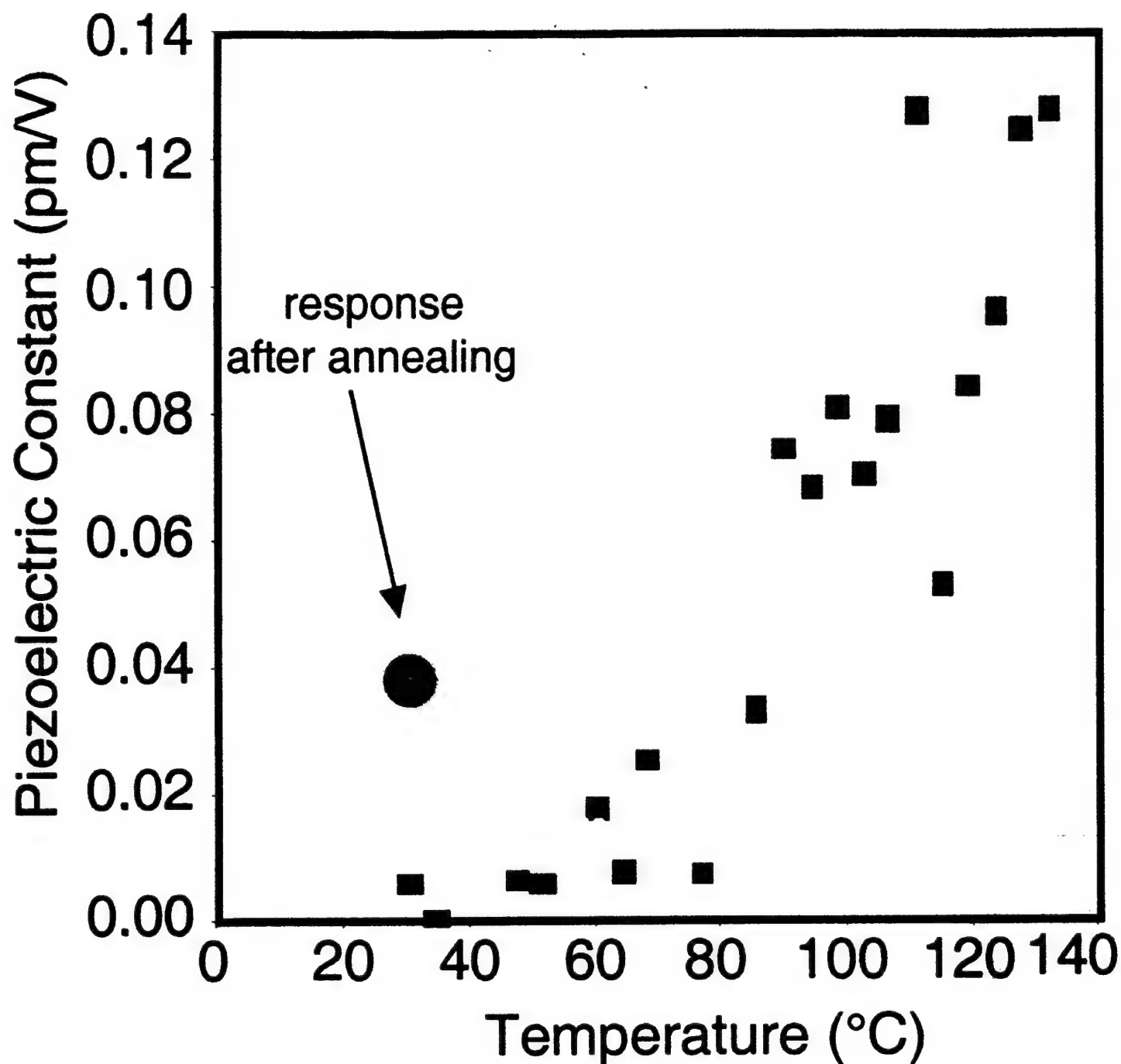


Figure 9. The piezoelectric response of the rodcoil film increases during annealing. Upon cooling to room temperature the piezoelectric properties remain non zero.

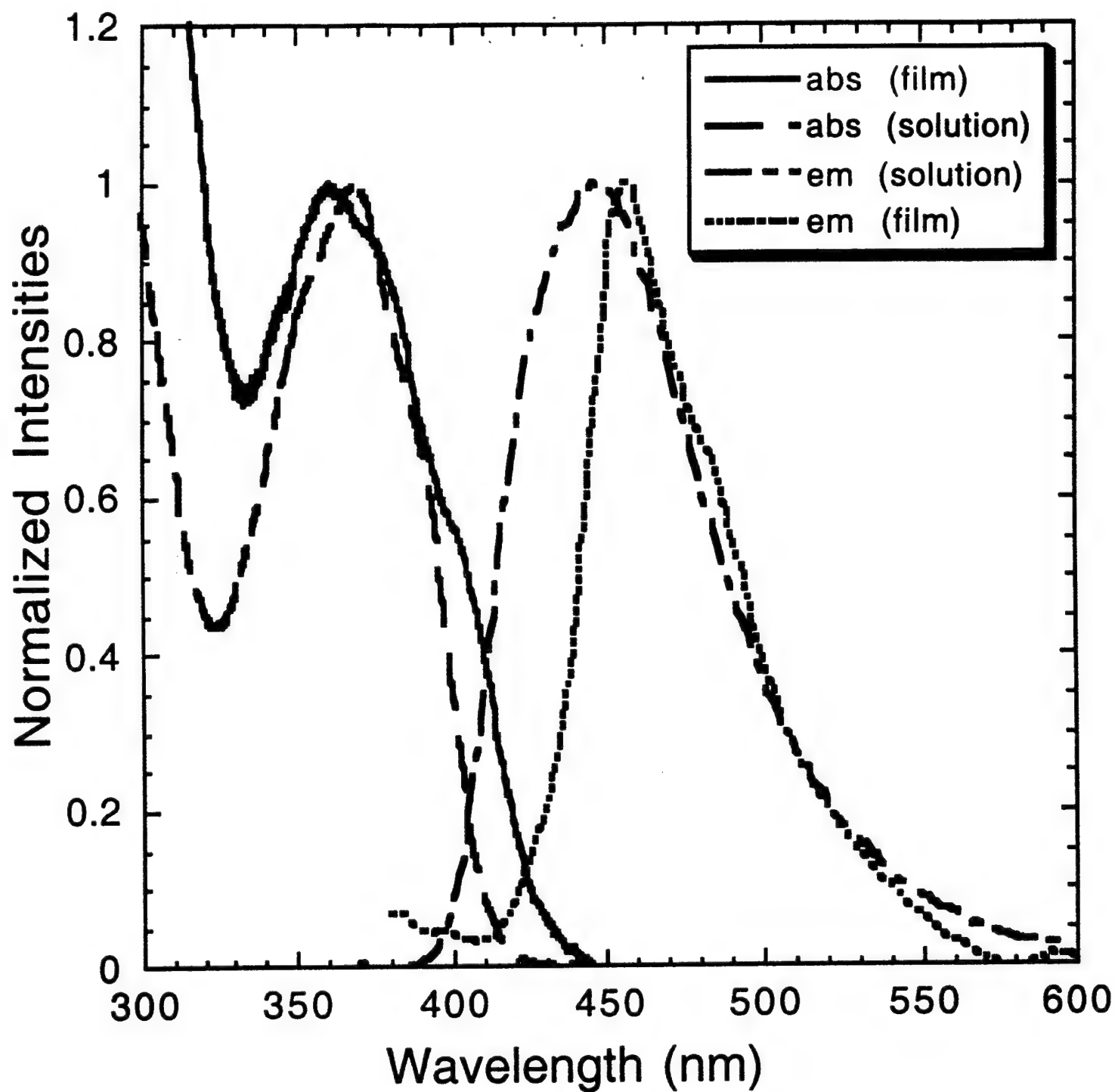


Figure 10. Nanostructured materials exhibiting interesting photonic behavior not typically observed in phenylene vinylene chromophores.

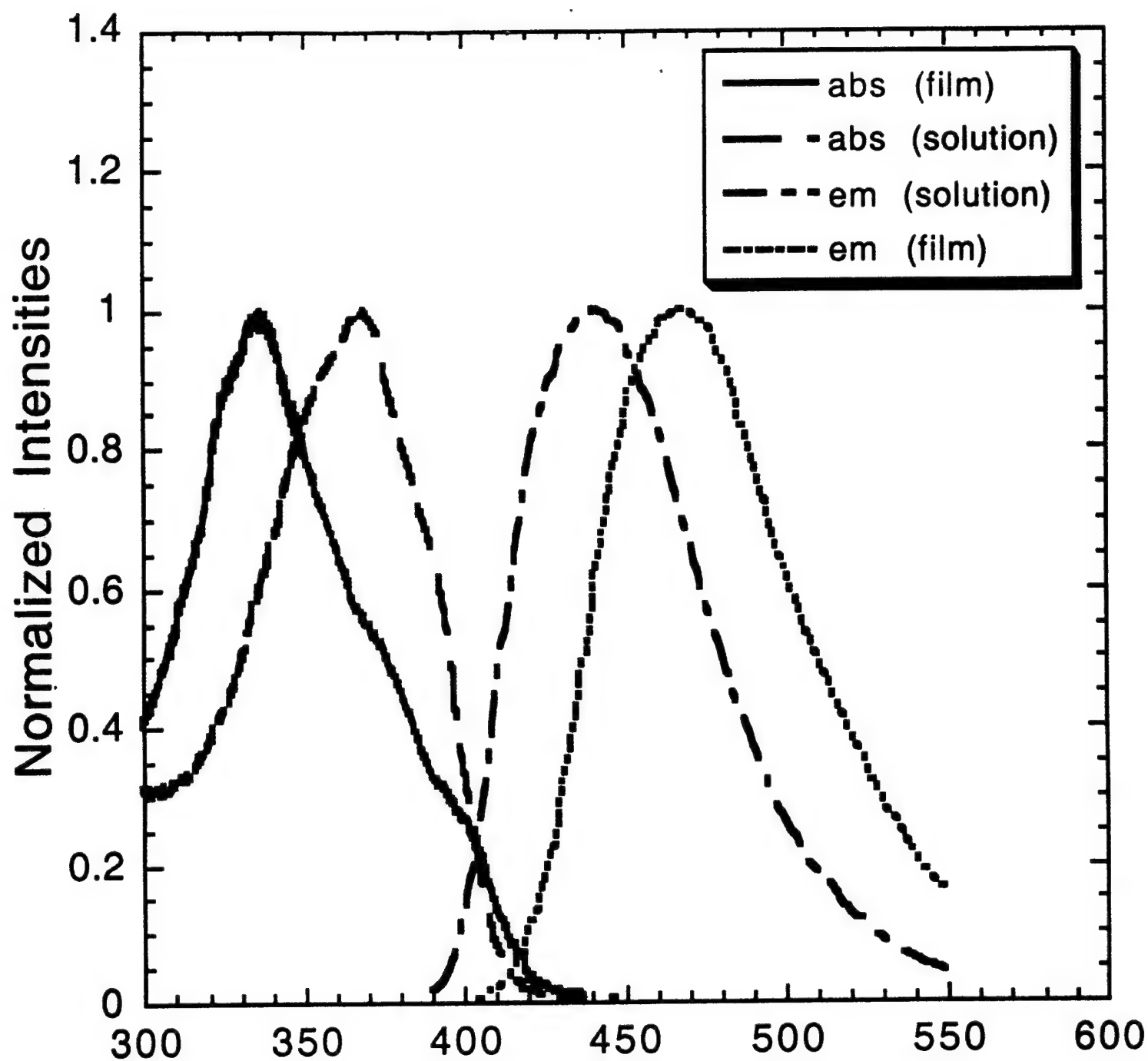


Figure 11. Large shift in absorption and emission spectra is typical of phenylene vinylene chromophores.

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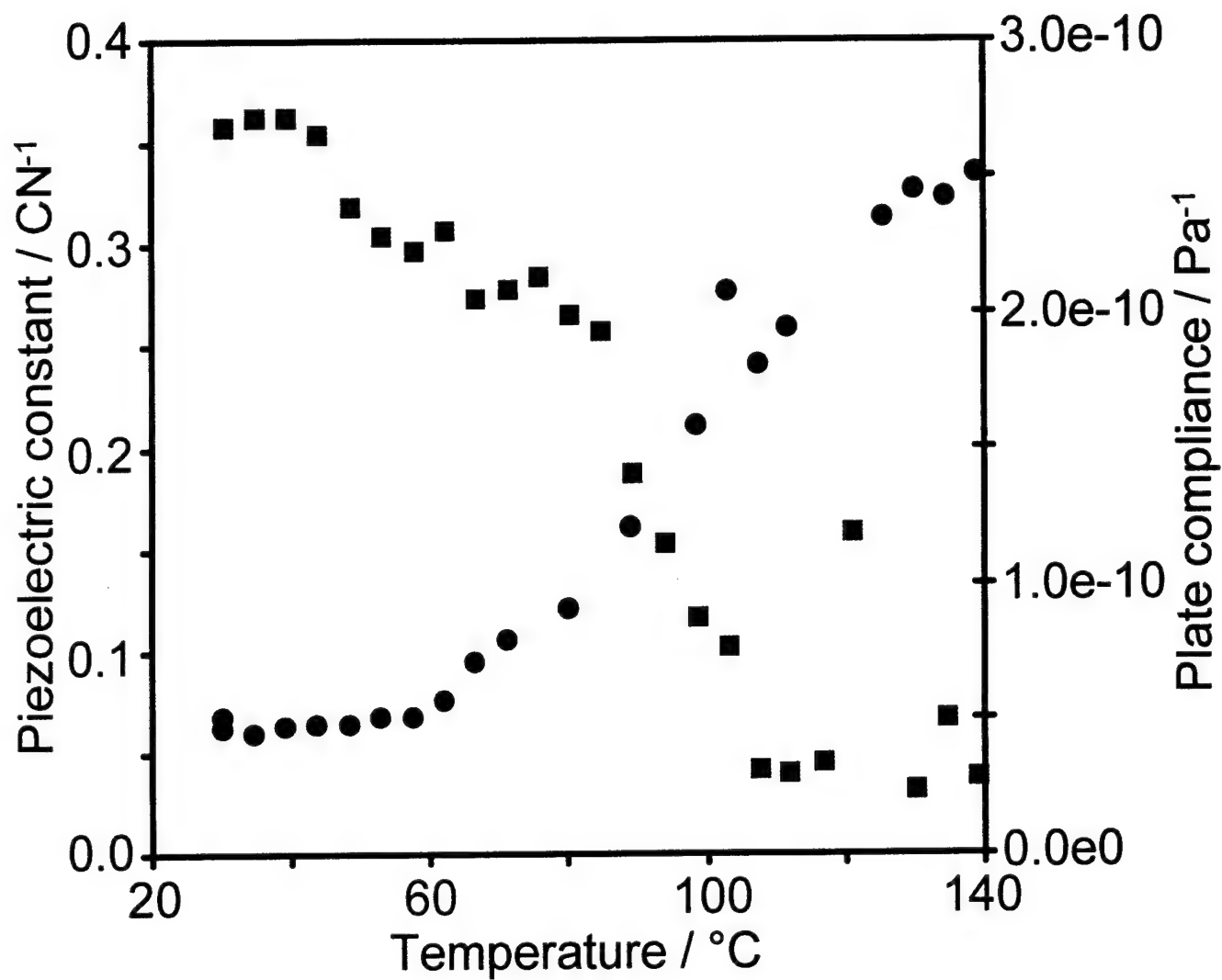


Figure 13. Decrease of piezoelectric activity of a poled sample and the corresponding increase in compressibility during the annealing procedure.

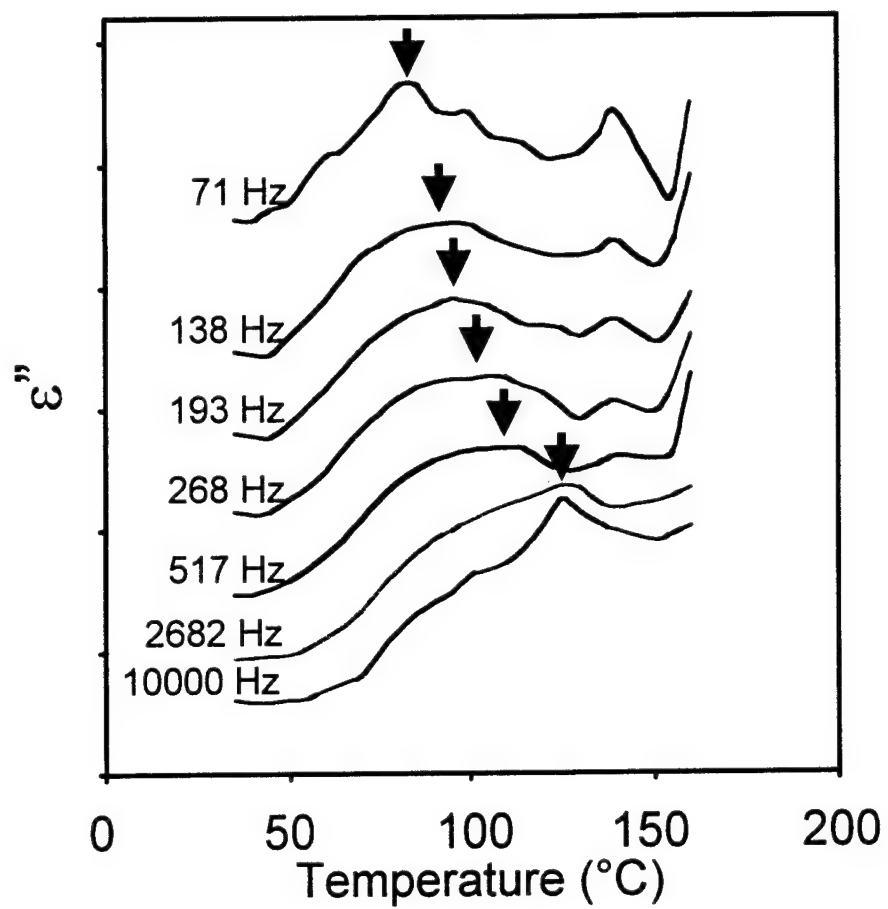


Figure 14. Dielectric spectroscopy demonstrates the occurrence of a molecular relaxation in the temperature range of 80-100 °C

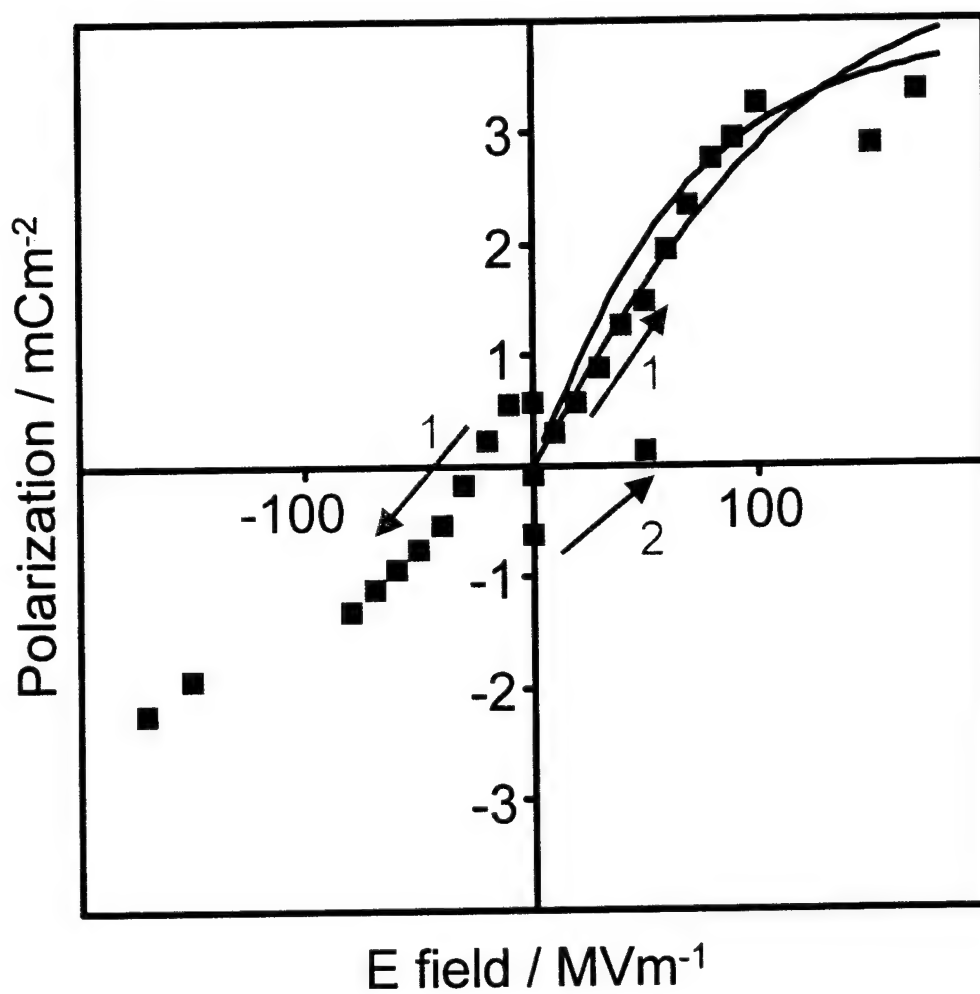
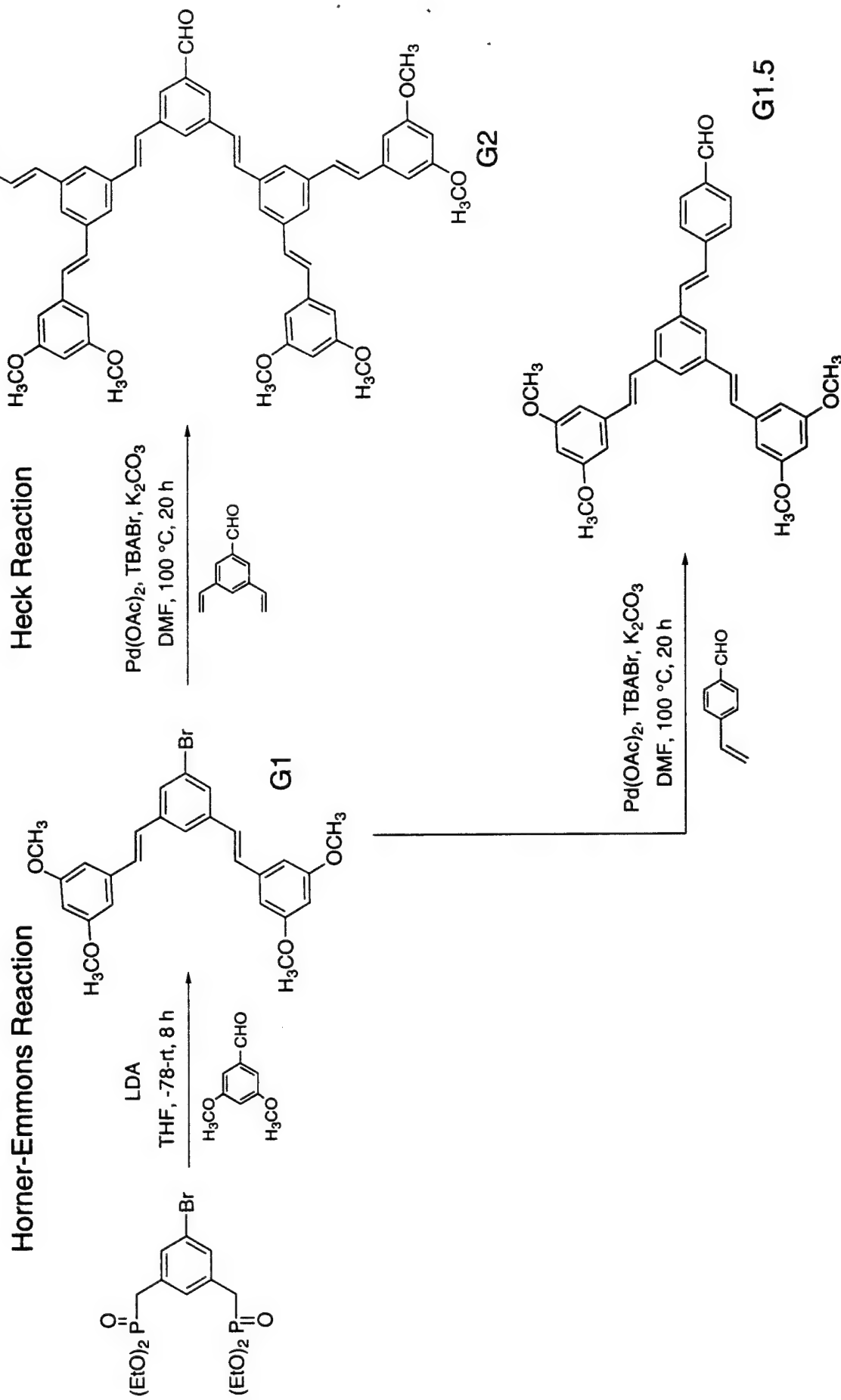
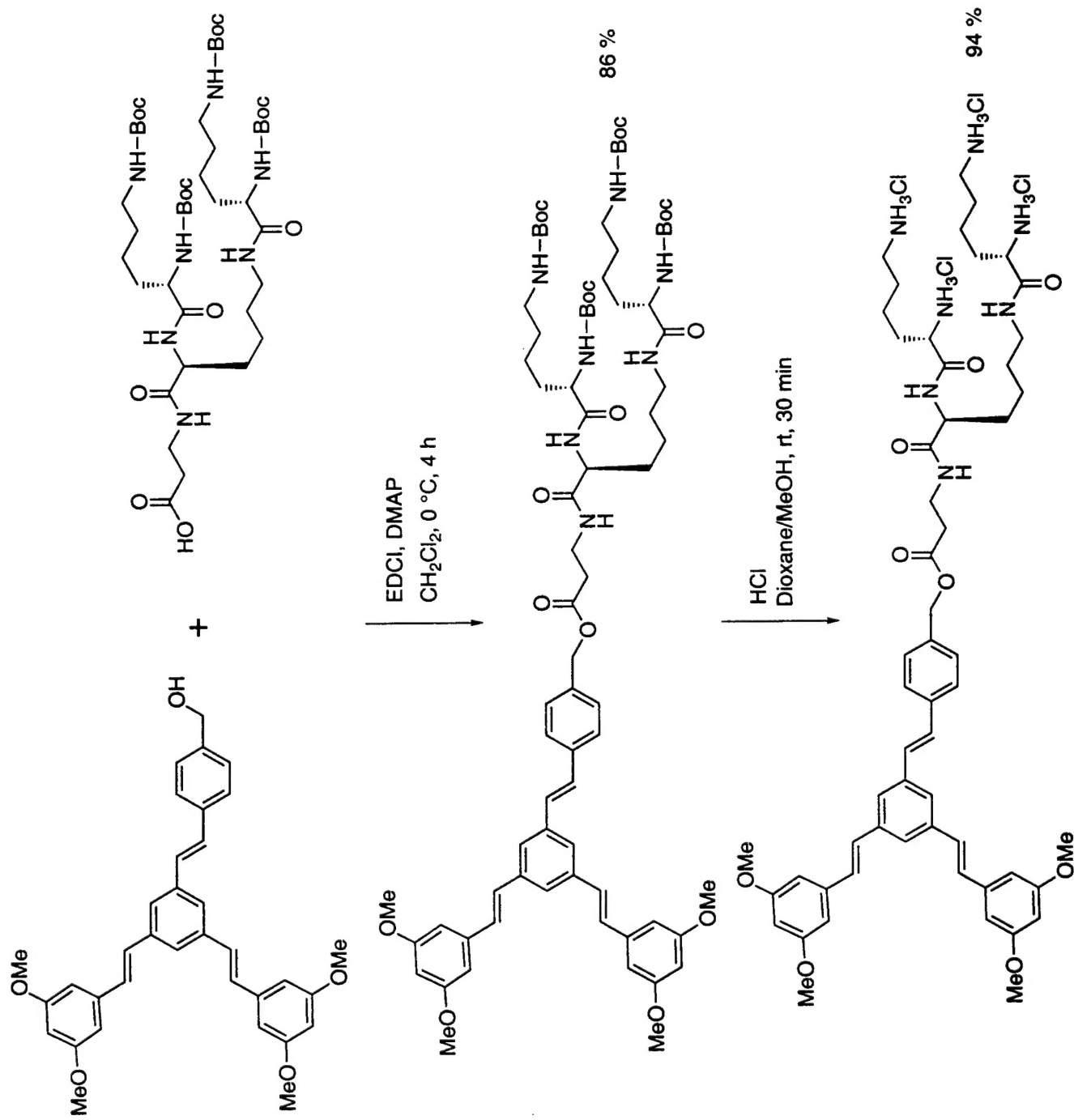


Figure 15. Hysteresis loop of a supramolecular rodcoil film indicating polarization saturation under both positive and negative bias. The remnant polarization is of the same order of magnitude as is observed for annealed samples.



Scheme 1. Orthogonal synthesis of phenylene vinylene dendrons. These dendrons are the luminescent, hydrophobic segments of novel amphiphiles.



Scheme 3. The synthesis of novel luminescent, branched amphiphiles.

Publications and Technical Reports

G. N. Tew, M. U. Pralle, and S. I. Stupp: Supramolecular Materials with Electro-Active Chemical Functions, *Angewandte Chemie*, **112**, 3, 527 (2000).

M. U. Pralle, K. Urayama, G. N. Tew, D. Neher, G. Wegner, and S. I. Stupp: Piezoelectricity in Polar Supramolecular Materials, *Angewandte Chemie*, **112**, 8, 1546 (2000).

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G.N. Tew, and S. I. Stupp, "Multifunctional Supramolecular Materials", *ACS Symposium Series: Functional Polymers*, A. O. Patil, D. N. Schulz, and B. Novak, eds., **218** (1998).

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Patents

U. S. Patent Application, Self-Assembling Compounds and Use of the Same to Induce Order in Organic Media, filed 9/99.